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HIGH ENERGY COLLIMATING FINE GRIDS FOR HESP PROGRAM FINAL REPORT

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HIGH ENERGY COLLIIMATING FINE GRIDS FOR HESP PROGRAM

1. INTRODUCTION

There is a need to develop fine pitch x-ray collimator grids as an enabling technology for planned future NASA missions. The grids consist of an array of thin parallel strips of x-ray absorbing material, such as tungsten, with pitches ranging from 34 microns to 2.036 millimeters. A sketch of such a grid is shown in Figure 1. The grids are the key components of a new class of spaceborne instruments known as "x-ray modulation collimators". These instruments are the first to produce images of celestial sources in the hard x-ray and gamma-ray spectral regions. For missions such as NASA's High Energy Solar Physics (HESP) mission, fine pitch grids designed to withstand the launch and on-orbit environments are required.

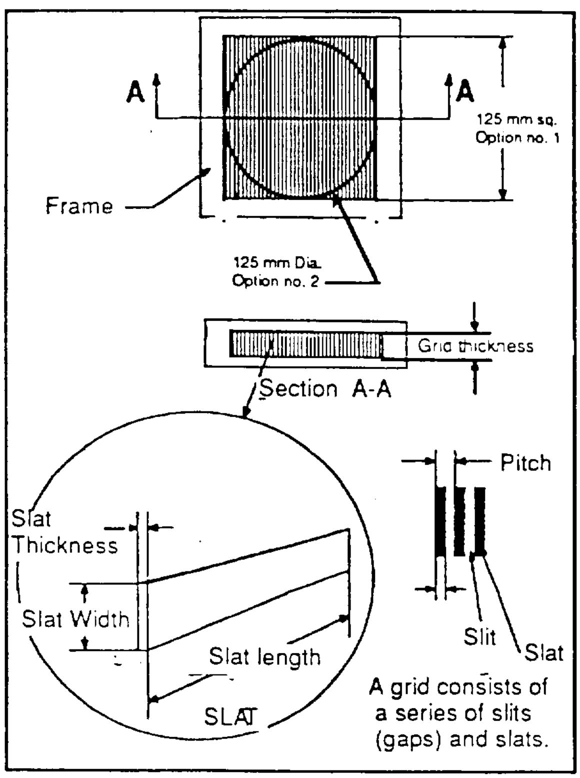


Figure 1: X-Ray Collimator Grid Terminology

The primary instrument on the first HESP flight is the High Energy Imaging Spectrometer (HEISPEC) which utilizes twelve rotating modulation collimators to image the sun at x-ray and gamma-ray wavelengths. A modulation collimator consists of a pair of aligned identical grids separated by a collimator tube, an HPGe or Si detector and on-board electronics to process and store the signal. Identical pairs of collimator grids, when rotated in front of Ge and Si detectors, impose the spatial modulation necessary for the HESP instrument to reconstruct an image of the sun. The spacecraft is spin stabilized in the equatorial plane with a spin rate of 15 rpm about the sun-pointing spin axis. It is this rotation which provides the modulation. HEISPEC incorporates twelve pairs of collimator grids whose pitches range from 34 microns to 2.036 mm. A table of the current HEISPEC Grid dimensions is shown in Appendix A.

TRW designed a pair of 4-inch diameter fine grids consisting of thin tungsten slats positioned in a crystal silicon metering structure which provided the required alignment and periodicity. The ends and a portion in the middle of each slat would be completely contained within the slot by the metering structure. The metering structure itself is chemically etched from a single monolithic silicon wafer using standard photolithographic etching techniques developed by the semiconductor industry. These techniques can be used to make structures with an absolute dimensional accuracy of much less than 1 micron. These grids were designed to meet the following goals:

DESIGN GOALS

Shape:..Circular aperture
Aperture:..69 mm
Grid thickness:.....................................1.75 mm
Grid pitch:..60 microns
Slat width:..25 microns
Slit width:..35 microns

A schematic drawing of the silicon metering structure design is shown in Figure 2. The tungsten slats are not shown. The tungsten slats function as x-ray and gamma-ray absorbers. However, since silicon is a low-Z material, its cross section to x-rays and gamma-rays is much lower than that of tungsten in the energy range of interest to HESP. Therefore, a silicon comb can be placed directly over the aperture of the grids with very little loss of throughput. This silicon comb serves to support the slats and discourage vibration or deformation.

The tungsten slats were chemically etched from tungsten foil sheets. Uniformity is very important in this application since an overly thick slat would not fit and might cause breakage. Overly thin slats were not expected to be a problem. Chemical etching was chosen to minimize the possibility of burrs or beads on the edges of the slats.

After the slots were etched into the silicon wafer and the tungsten slats fabricated, the slats were to be carefully inserted into the slots. Then a silicon cover ring (with bridge) would be placed over the metering structure, capturing the ends and center sections of the slats.

The various components of grid fabrication are discussed in the following sections.

2. THE SILICON METERING STRUCTURE

2.1 Etching techniques

The major issue is the etching of the high aspect ratio slots to the accuracy required for the metering structure. Deep slots are etched in silicon by letting KOH etchant etch through a narrow slot made in the silicon nitride layer. The slot is made in the silicon nitride layer by the following standard photolithographic technique: Photoresist is placed over the silicon nitride layer and a photomask containing the pattern to be etched is placed over the photoresist. The photoresist is exposed through the mask and developed, leaving areas of the silicon nitride open and vulnerable to plasma etching which is the next step. In plasma etching, the silicon nitride unprotected by photoresist is removed, transferring the pattern (in this case, narrow slits) to the silicon nitride layer, thereby exposing the silicon wafer below. After plasma etching, the remaining photoresist is chemically removed.

The depth and width of the slot in the silicon is determined by the etch rates in the two different directions as shown in Figure 3 The vertical direction is the [110]direction, and its etch rate is r[110]. The horizontal direction is the [111] direction and its etch rate is r[111]. The ratio of the etch rates along the two crystal planes gives the final slot depth to slot width ratio, i.e., r[110]/r[111]=W/U, where W is the desired slot depth and U is the undercut of silicon nitride layer. As the horizontal etching proceeds, the width of the slot grows and the silicon nitride layer is undercut. It has been established experimentally that, as this undercutting proceeds, the walls of the slot remain vertical.

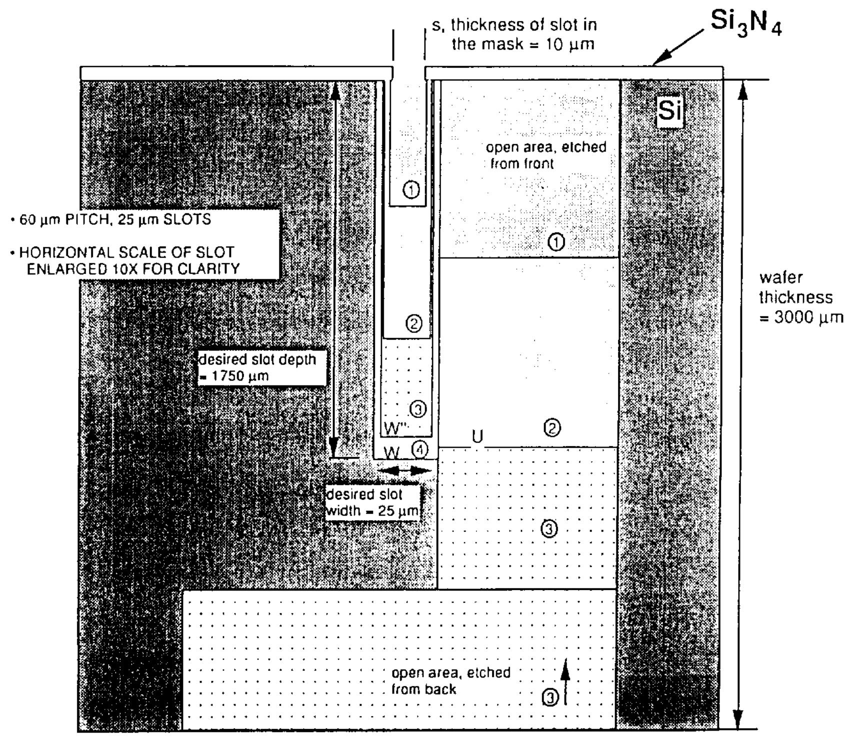


Figure 2: Schematic of Four-Step Process For Etching High Aspect Ratio Slats in Silicon. (Note: horizontal scale expanded by factor of 10.)

W/U is a function of the deviation of the slot from the true crystalline orientation. Actually two components of the orientation are involved. One is the off-orientation of the wafer surface from the [110] plane and the other is the off-orientation of the mask pattern from the intercept of the [111] plane with the wafer surface. If T is the desired slot width and S is the width of the slot in the silicon nitride coating and U is the undercut to the silicon nitride layer, then

$$T = S + 2U.$$

If Q is defined in units of minutes as the off-orientation angle of the mask pattern slots from the intercept of the [111] plane with the wafer surface, then Kendall (1) has shown that

$$W/U = 2100/Q(min).$$

Kendall(1) has also shown that W/U is also a function of the concentration of the KOH etchant and that the absolute value of the [110] etch rate is a function of KOH concentration and temperature. When etching particularly deep slots, the temperature of the KOH must be maintained in the region of 70 to 85 degrees C in order to keep the total time required for etching down to reasonable values.

Given the required geometry, the dimension, S, of the slot opening in the silicn nitride which is required to achieve the requisite final slot dimensions, T and W, may be calculated for a known off-orientation. The greater the off-orientation Q, the narrower will be the required slot opening S. For S to be greater than zero, the following relationship must be satisfied:

$$Q < 1050\ T/W.$$

In the current design, $T = 25$ microns and $W = 1750$ microns which forces the requirment that the off-orientation Q be less than 15 minutes. In the end, this requirement proved to be the most difficult to meet.

The final dimensions are very sensitive to even small differences in the off-orientation Q. When the slot is etched to the desired depth, it will either be too narrow or too wide depending upon whether the assumed off-orientation is less than or greater than the actual misorientation.

Finally, it should be noted that Kendall has also determined that the ratio of the etch rate high aspect slots to the etch rate of open areas is 0.7.

Further discussions of the technical issues associated with etching of silicon at high aspect ratios may be found in Appendices B and C.

2.2 Description of the Wafers

A total of nine four-inch diameter silicon crystal wafers 3 millimeters thick were purchased through International Wafer Service. The wafers were marked with the order in which each was removed from the boule (serial number) on the topside. The wafers were lapped and polished and the crystal orientations provided by Recticon. Then the wafers were coated on both sides with 2500 Angstroms of low-stress silicon nitride.

The surface off-orientation with the [110] plane of each wafer is measured via x-ray diffraction patterns and recorded. The off-orientations are 4 minutes in this case. The off-orientation of the intersection of the [111] plane with a feature of the silicon (in this case, a flat edge of the otherwise round wafer) was also measured and was reported by Recticon to be either 10 or 11 minutes, depending on the serial number of the wafer. The rotational sense of the angle was also specified.

The wafers were expected to be delivered 24 August 1993. However, they were not delivered until 17 September 1993, 3 1/2 weeks later. This delay in delivery initiated serious time pressure, since delivery was then scheduled to be in October.

2.3 The silicon metering structure design

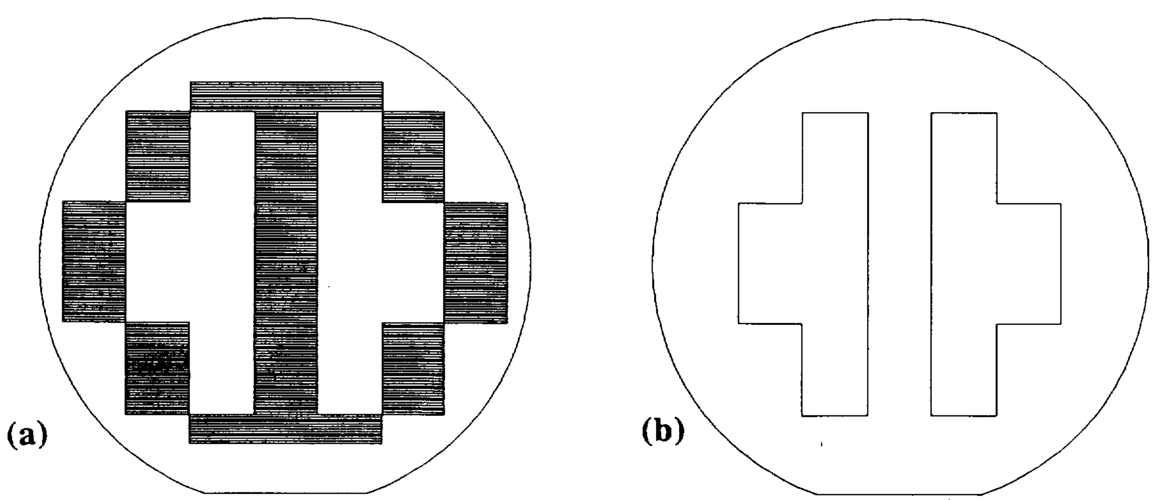


Figure 3: Sketches of the Top (a) and Back (b) Photomasks To Be Used In Etching the Silicon Metering Structure.

The photomasks for etching the silicon metering structure are shown in Figure 3. The top photomask defines the slots that will hold the tungsten slats. It also contains large open areas in order to leave a minimum of silicon directly over the aperture. The slots in this mask are narrower than the width of the desired slots in the silicon wafer to allow for the undercutting that will occur. The slots are etched down to a depth which will just contain the tungsten slats, although the precise value of this depth is not critical. The open areas on the front are etched at the same time as the slots. It is important to note at this point that the open areas etch faster than deep slots by a factor of 10:7. Even with this faster etching, the open area will not be etched all the way through the 3 millimeter wafer by the time the etching must be stopped. Therefore, the open areas are etched from the back side as well. The etching is begun using only the top photomask. The back photomask is exposed on the back side at the point such that when the open areas are eched from both sides, they will meet when the slots have been etched down to the desired depth.

2.4 Fabrication

Initially wafer 001 was coated with photoresist. The top photomask was aligned with a 10 minute clockwise rotation relative to the flat edge of the wafer. Based on the certification provided, this arrangement should have corresponded to the minimum off-orientation of 4 minutes. The photoresist was exposed and developed. The silicon nitride layer was plasma etched and the wafer was immersed in 44% KOH at 85 degrees C for 5 hours. In these conditions, the open area etching rate is 2 microns/minute. The predicted slot depth is 420 microns. (When the wafer was subsequently sacrificed and the depth measured, this proved to be a close estimate.) However, the observed undercutting, U, was 6 microns. Using Kendall's expression, the off-orientation was calculated to be 31.5 minutes.

Wafer 009 was then coated with photoresist, and the top photomask was aligned with a 10 minute counter-clockwise rotation relative to the flat edge of the wafer. The results of etching 009 were expected to be either much better or much worse than those of 001. After the same procedures, wafer 009 was examined and found to be entirely similar to 001. This led to the conclusion that we were dealing with a process-related problem, probably depletion of the KOH in the bottom of the slots, which was causing the increased rate of undercutting. Also Recticon was asked to examine their records, looking for any information which might have a bearing on the problem. Nothing was found.

Wafer 002 was processed using the 10 minute clockwise rotation relative to the flat edge of the wafer. During the KOH etching phase, the wafer was removed from the solution every hour, cleaned, dried and photographed. After each removal, the wafer was wiped gently to remove the undercut silicon nitride layer. There was no dramatic increase of the ratio of the etching rates r[110] to r[111].

At this point, due to deadline considerations, it was decided to etch wafers 003 and 004 as deeply as possible while still maintaining the integrity of the slot walls.

In reality, there are two intersections of [111] planes with the surface of the wafer. The second intersect is 70.5 degrees from the first intersect which is nearly parallel to the slots. While wafers 003 and 004 were in process, wafers 001 and 009 were sacrificed by cleaving them along the second [111] intersect . Wafer 009 cleaved cleanly. Wafer 001 began to cleave similarly, but the cleavage plane was ragged and veered off. Wafer 001 was "flipped" relative to 009. When the wafers were rotated in opposite directions, the rotations brought the mask alignments into exactly similar orientations with respect to the intersection of the [111] plane of that wafer. Hence, similar results were inevitable.

After sacrificing the wafers, photographs indicated that the walls of the slots were parallel, but due to the high level of off-orientation, the planes of the slot walls were not completely smooth. The bottoms of the slots were found to be smooth.

Careful measurements of the angles of the slot pattern relative to the flat side of the wafer and the second [111] cleavage plane indicate that the true crystal orientation differs from that stated in the certification by an additional 10 to 12 minutes, bringing the total off-orientation to about 33 minutes. The etching rate ratios observed are entirely appropriate for that amount of off-orientation. Wafers 003 and 004, if measured, would also be off by that amount.

2.5 The Delivered Metering Structures

The metering structures finally delivered were a time-driven compromise which appeared to have a good possibility of fulfilling the minimal requirements of holding the tungsten slats with the required spacing. Since the open areas were deeper than the slots, there would be no interference with the lower edges of the slats. The slots were somewhat wider than the original design called for, but the extra width was expected to make the assembly task easier. The parameters describing the two metering structures delivered are shown below:

	s/n 003	s/n 004
wafer thickness (unetched)	3021 microns	3018 microns
thickness in etched open areas	1867 microns	1844 microns
thickness of material removed	1154 microns	1174 microns
calculated slot depth	808 microns	822 microns
slot width (measured)	36 microns	33 microns

The calculated slot depth of approximately 800 microns will at best contain only 46 % of the 1750 micron wide slat, leaving approximately 55% extending above the surface of the wafer.

3. TUNGSTEN FOIL

3.1 Requirements

Each grid required approximately 1270 thin rectangular slats. The slats were rectangles chemically cut from off-the-shelf tungsten foil 1.0 ±- 0.1 mil (25.4 ± - 2.5 microns) thick. Each grid required 3 slat lengths in the following numbers and lengths:

212 slats which were 1.15" in length
634 slats which were 2.15" in length
424 slats which were 3.15" in length

It should be noted that these numbers are in the ratio 1:3:2. All slats were 1.750 millimeters wide.

3.2 Fabrication steps

A vendor was found which could provide off-the-shelf foil sheets with the required thickness and tolerances. The vendor provided us with a sample sheet for measurement. The thickness and variations were within our specifications. Nine 6" by 20" sheets were purchased. These were measured and all were found to be suitable. Average thicknesses and standard deviations for each sheet were calculated from the sets of measurements. The sheets were observed to be both stiff and brittle.

A vendor was found to chemically etch the slats from the tungsten sheets. Each sheet was required to produce a proportional number of slats in each length. Furthermore, we required that sheet identification be maintained so that, if necessary, we could use the thicker or thinner sheets preferentially. When examined under a microscope, the tungsten slats appeared to be cleanly cut and free of burrs or beads.

4. ASSEMBLY

In the design phase, little difficulty was expected in the assembly of the tungsten slats into the silicon metering structure. Ideally the slots would be a minimum of 1750 microns deep and approximately 28 microns wide. First the silicon nitride layer would be removed from the wafer. Then each individual slat would be held by one of its tabs remaining after tungsten etching, eased into position and then "patted" into place. Ideally the slat would be totally contained within the slot except in the open areas.

In practice, assembly proved quite difficult. Since the slots were only about 800 microns deep, the slats would be at best only about 45 % contained in the slot, not totally contained within the slots as the original design required.

Initially difficulties were encountered in placing the slats in the slots. Although the slots appeared to be clean, it was impossible to position the slats firmly in the slots. Because the slats were less than 40 % contained in the slots, the assembly technicians working with the finest tweezers could not position a slat without touching the neighboring slat(s) which were dislodged by the merest touch.

Also considerable breakage was occurring in the metering structure. The tungsten slats were sufficiently rigid that movement within the slot during positioning caused the slot walls to break. Since this design depends on the uniformity of the foil slats (the x-ray and gamma-ray absorbers) which was maintained, some breakage of the support walls was not too serious.

After a number of hours in which only a relatively small number of slats were successfully positioned, these slats were epoxied into place using a low-temperature-curing epoxy. This was not the original design, but use of the cover ring was out of the question in this situation.

The grids were then packaged for shipping and sent by counter-to-counter air shipping for subsequent delivery to GSFC.

5. REFERENCE

(1) ORIENTATIONS OF THE THIRD KIND: THE COMING OF AGE OF (110) SILICON, D.L. Kendall and G.R. de Guel, in "Micromachining and Micropackaging of Transducers", edited by C. D. Fung et.al., Elsevier Science Publishers, Amsterdam, 1985 (pp. 113-130).

Appendix A

HEISPEC Grid Dimensions 28 April 1993

Collimator Length (mm)	1750.
Bottom collimator diameter (mm)	71.0
Top collimator diameter (mm)	89.3
Angular overlap (degrees)	0.300
Ratio of slit width to pitch	0.600

Collimator	1	2	3	4	5	6	7	8	9	10	11	12
FWHM Resolution (arcsec)	2.00	2.90	4.21	6.11	8.86	12.86	18.66	27.08	39.29	57.00	82.70	120.00
Pitch (mm)	0.034	0.049	0.071	0.104	0.150	0.218	0.317	0.459	0.667	0.967	1.403	2.036
Slit width (mm)	0.020	0.030	0.043	0.062	0.090	0.131	0.190	0.276	0.400	0.580	0.842	1.222
Thickness (mm)	1.17	1.69	2.46	3.56	5.17	7.50	10.00	10.00	10.00	10.00	10.00	10.00
Field of View (deg.)	1.00	1.00	1.00	1.00	1.00	1.00	1.09	1.58	2.29	3.32	4.81	6.97
Grid rim thickness (mm)	10.00											
Grid density (g/cm^3)	19.30											
Top grid weight (kg)	0.09	0.13	0.19	0.27	0.40	0.58	0.77	0.77	0.77	0.77	0.77	0.77
Total (kg)	6.28											
Bottom grid weight (kg)	0.06	0.09	0.13	0.19	0.28	0.40	0.54	0.54	0.54	0.54	0.54	0.54
Total (kg)	4.37											
Energy for 50.% transmission												
Energy (keV)	308.	379.	481.	648.	947.	1664.	0.	0.	0.	0.	0.	0.
Diffraction Energy limit (keV) with 10%, 50%, and 100% reduction in modulation amplitude												
(0.0 = < .1 keV)												
10% red, @ fundamental	13.1	6.2	3.0	1.4	0.7	0.3	0.2	0.0	0.0	0.0	0.0	0.0
10% red. @ 2nd harmon.	52.5	24.9	11.8	5.6	2.7	1.3	0.6	0.3	0.1	0.0	0.0	0.0
10% red. @ 3rd harmon.	118.1	56.1	26.6	12.7	6.0	2.9	1.4	0.6	0.3	0.1	0.0	0.0
50% red. @ fundamental	5.7	2.7	1.3	0.6	0.3	0.1	0.0	0.0	0.0	0.0	0.0	0.0
50% red. @ 2nd harmon.	22.6	10.7	5.1	2.4	1.2	0.5	0.3	0.1	0.0	0.0	0.0	0.0
50% red. @ 3rd harmon.	50.9	24.2	11.5	5.5	2.6	1.2	0.6	0.3	0.1	0.0	0.0	0.0
100% red. @ fundamental	3.8	1.8	0.9	0.4	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0
100% red. @ 2nd harmon.	15.1	7.2	3.4	1.6	0.8	0.4	0.2	0.0	0.0	0.0	0.0	0.0
100% red. @ 3rd harmon.	33.9	16.1	7.7	3.6	1.7	0.8	0.4	0.2	0.0	0.0	0.0	0.0
First Diffraction Peak in Modulation Amplitude (keV)												
1st pk, fundamental	1.9	0.9	0.4	0.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
1st pk, 2nd harmonic	7.5	3.6	1.7	0.8	0.4	0.2	0.0	0.0	0.0	0.0	0.0	0.0
1st pk, 3rd harmonic	17.0	8.1	3.8	1.8	0.9	0.4	0.2	0.0	0.0	0.0	0.0	0.0

Interoffice Correspondence

TRW Space & Technology Group

TRW

Subject:	Date:	From:
Undercutting during silicon etching	7/15/93	E. N. Frazier E.F.
To:	cc:	Location/Phone:
File	GSFC	R9/1082 x20282

Deep slots are etched in silicon by letting KOH etchant etch through a narrow slit made in the silicon nitride layer which is made in the photomask process. The depth and width of this slot is determined by the etch rates in the two different directions (see figure 1) The vertical direction is the [110] direction, and its etch rate is $r_{[110]}$. The horizontal direction is the [111] direction and its etch rate is $r_{[111]}$. The ratio $r_{[110]}/r_{[111]}$ is D/U. As the horizontal etching proceeds, the width of the slot grows and the silicon nitride layer is undercut. It has been established empirically that, as this undercutting proceeds, the walls of the slot remain vertical.

D/U is a function of the deviation of the slot from the true crystalline orientation. Actually two components of the orientation are involved. One is the off-orientation of the surface from the [110] plane and the other is the off-orientation of the mask pattern from the intercept of the [111] plane with the surface. We can define off-orientation as the angle between the [11$\bar{2}$] direction and the pattern edge lying on the silicon surface, equal to θ (in minutes). We define t as the desired slot width, S as the width of the slit in the silicon nitride coating, and W as the desired slot depth. We can write therefore:

$$t = S + \frac{2W}{D/U}$$

Now Kendall has shown that D/U = 2100/θ(min.). (See figure 2) . Kendall has also shown that D/U is also a function of the concentration of the KOH etchant, as can be seen in the figure 3. Figure 4, also from Kendall, shows the absolute value of the r[110] etch rate as a function of KOH concentration and temperature. It shows that, when etching particularly deep slots as we are doing, the temperature of the KOH must be maintained in the region of 70 to 85 °C in order to keep the total time required for etching down to reasonable values.

We are considering two geometries:

I. t = 25 μm

W = 1750 μm

II. t = 40 μm

W = 3000 μm

Substituting these t and W values into the above equation will enable us to calculate the dimension, S, the slit opening in the silicon nitride required to achieve the requisite slot dimensions, t & W, for a particular off-orientation. We note that the greater the off-orientation, θ, the narrower will the required slit opening, S, be. In fact, for S to be greater than zero, we must satisfy the relationship,

$$t > \frac{2W}{D/u} \quad \text{or}$$

$$\theta < \frac{2100}{2W} \cdot t$$

for geometry I, $\theta < 15$min; for geometry II, $\theta < 14$ min.

We now explore the means of achieving the desired dimensions, t and W for geometry II. If we could measure the misorientation very precisely, we could theoretically choose the slit opening, S, which would give us the required dimensions, t and W. However, we must assume a value θ', which is not exact. We recognize that the final dimensions are very sensitive to even small differences in the misorientation θ, and that when we have etched the slot to its desired depth, we will find the slot either too narrow or too wide depending upon whether our assumed misorientation, θ' is less than the actual misorientation, θ, or greater than it.

One possible approach is based on the recognition that the dimensions of the slot <u>width</u> are critical to holding the tungsten slats parallel, but that we can tolerate some variation in slot <u>depth</u> from the target value of W. The final slot depth may be somewhat smaller or greater than the target value, W. In this approach, we can simply assume a misorientation, θ', choose the corresponding mask slit width, S, and then etch until the desired slot width, t, is achieved. We can thus calculate for example the slot depth, W', required to achieve the necessary slot width. With this approach, the question becomes, How much does W' deviate from W for anticipated values of θ and θ'?

The table below gives the values of D/U, t-S, and S as a function of θ for geometry II, t = 40 μm.

θ, min.	D/U	t-S, μm	S, μm
1	2100	2.86	37.14
2	1050	5.71	34.29
3	700	8,57	31.43
4	525	11.43	28.57
5	420	14.29	25.71
6	350	17.14	22.86
7	300	20.0	20.0
8	262.5	22.86	17.14
9	233	25.75	14.25
10	210	28.57	11.43
11	191	31.41	8.59
12	175	34.29	5.71
13	161.5	37.15	2.85
14	150	40.0	0

We have available silicon wafers, 4 inches in diameter and 0.164 inches thick. the surface misorientation with the [110] plane of each wafer has already been measured via x-ray diffraction patterns and marked on the wafer. These misorientations are in the range from 1 min. to 5 min. Now we need to measure the orientation of the intersection of the [111] plane with the wafer surface so we can orient the mask pattern properly. This will be done by doing a preliminary etch with an orientation fan pattern: A small pattern of individual slits will vary in orientation from one another by one minute of arc, forming a fan pattern. After this preliminary etching we will examine the etched slots for undercutting. The minimum undercutting will correspond to the least misorientation. We believe that we can target this misorientation, θ', to ± 2 min. of the true misorientation. So the total anticipated misorientation, q, of our current 4 inch wafer set should be in the range of 3 to 7 minutes. It is important to note that the surface orientation of the existing 2 inch wafers has not been measured, and in fact is probably larger than the 1 to 5 minutes of the 4 inch wafers, since considerable care was put into lapping the 4 inch wafers. Therefore any results that we might get from etching the 2 inch wafers will probably not be applicable toward the 4 inch wafers.

The following is a sample calculation for the case where the targeted misorientation is 8 min., but the actual misorientation differs from this value, θ' by 1, 2, and 3 minutes more and by 1, 2, and 3 minutes less. We want to calculate W' = the slot depth resulting etching until slot width t reaches 40 μm when the mask slit width is based on the assumption θ' = 8 min., but the actual misorientation, θ, which determines D/U is greater or less.

$$W' = \frac{(t-S)}{2} \cdot \frac{D}{U}$$

From the table, we have

$\theta' = 8$ min. - $t\text{-}S = 22.86\ \mu m$

if $\theta = 5$ min., for example, then from the table, the true D/U = 420.

then:

$$W' = \frac{22.86}{2} \times 420$$

$$W' = 4800\ \mu m$$

Similar calculations can be made for other values of θ', and θ-θ'. The results of these calculations are summarized on the graph on the figure 5. This graph shows that as the misorientation approaches the maximum permissible, i.e. 14 minutes, the effect of a difference between the targeted θ' and the actual misorientation, θ, becomes of less effect in preventing us from attaining our desired slot geometry. However the larger θ' values lead us to use an ever smaller mask slit, S, in the silicon nitride. This will have the undesirable side effect of isolating the KOH that is deep inside the slot from the general KOH solution. The result of this is that, as the etching proceeds, the concentration of KOH in the slot will decrease, and as can bee seen in figure 3, the etch ratio will change.

We appear to be caught between two unpleasant alternatives: On one hand, we can work with rather large misorientations, say 10 minutes or more. The actual slot depth W' will probably be reasonably close to the desired depth, W, but the process will be difficult to control because of the KOH depletion deep in the slot and in fact the etching might become quite slow. On the other hand, we can attempt to achieve very small misorientations, say 3 minutes or less, and we will be assured of W' being reasonably close to W. But this will require extremely careful initial lapping and measuring of the wafer. Clearly, we need to establish an etching process that is iterative in the sense that we can begin with reasonable misorientations, observe how the etching is proceeding, and then change the process appropriately so that we converge on the desired slot width and depth.

One possible iterative process is as follows: We carry out the etching in stages, and at the conclusion of each stage we will measure the dimensions of the slot. We can measure U, the undercut by the extension of the silicon nitride ledge over the slot. These measurements will give us an empirical value for D/U, characteristic of the actual misorientation of the particular silicon wafer. We can then change the etch ratio, r110/r111, if necessary by changing the KOH concentration. Initially we assume a particular etch ratio on the basis of the measured off-orientation of our fan pattern and design the slit in the silicon nitride to give us the desired slot width when the target slot depth is attained. If for example the actual ratio exceeds the assumed ratio, our slot width will be too narrow when we reach our target depth. By decreasing our KOH concentration for later etching steps, we will reduce the etch rate ratio, resulting in a greater slot width. In this approach, our choice of KOH concentration is based on etch rate ratio and not on etch rate, so we may be forced to increase the process time considerably. This approach will require much more time in general The iterative measurements have to be made which requires hand labor, and the etch rate may decrease.

There is another completely separate result of the undercutting effect, which is illustrated in the figure 6. At the end of a narrow slot, other [111] surfaces develop so that for a distance of 2W (where W is the slot depth) the "floor" of the slot is not flat. This part of the slot cannot be used for holding a tungsten slat. For our geometry II, W= 3000 μm, so this "unoccupied length of slot must be 6 mm on each side of the span. This decreases the open areas which can be used. This is illustrated in figure 7. This effect also happens to the combs which are intended to stabilize the tungsten slats. The edges lying in the [111] plane will be preserved. However the edges lying perpendicular to this plane will be attacked. Thus the comb will be attacked and undercut to a distance equal to the slot depth on both exposed sides. For geometry II, this is 3 mm on each side. We must increase the width of the pattern for the comb to compensate for this. Figure 7 shows masks required to compensate for both of these effects. Note that for our 4 inch diameter silicon wafer and 100 micron pitch grids (geometry II), we are now limited to a two inch aperture if we adopt a simple square aperture pattern. For geometry I and even finer pitches, this effect becomes less.

The general conclusion from all of these considerations about undercutting is that we have to include these effects very carefully in the design of the mask and in a specific step-by step procedure that will lead to the desired slot width and depth in a controlled way. That will be the subject of another memo.

UNDERCUTTING DURING SILICON ETCHING

(for 100μm pitch)

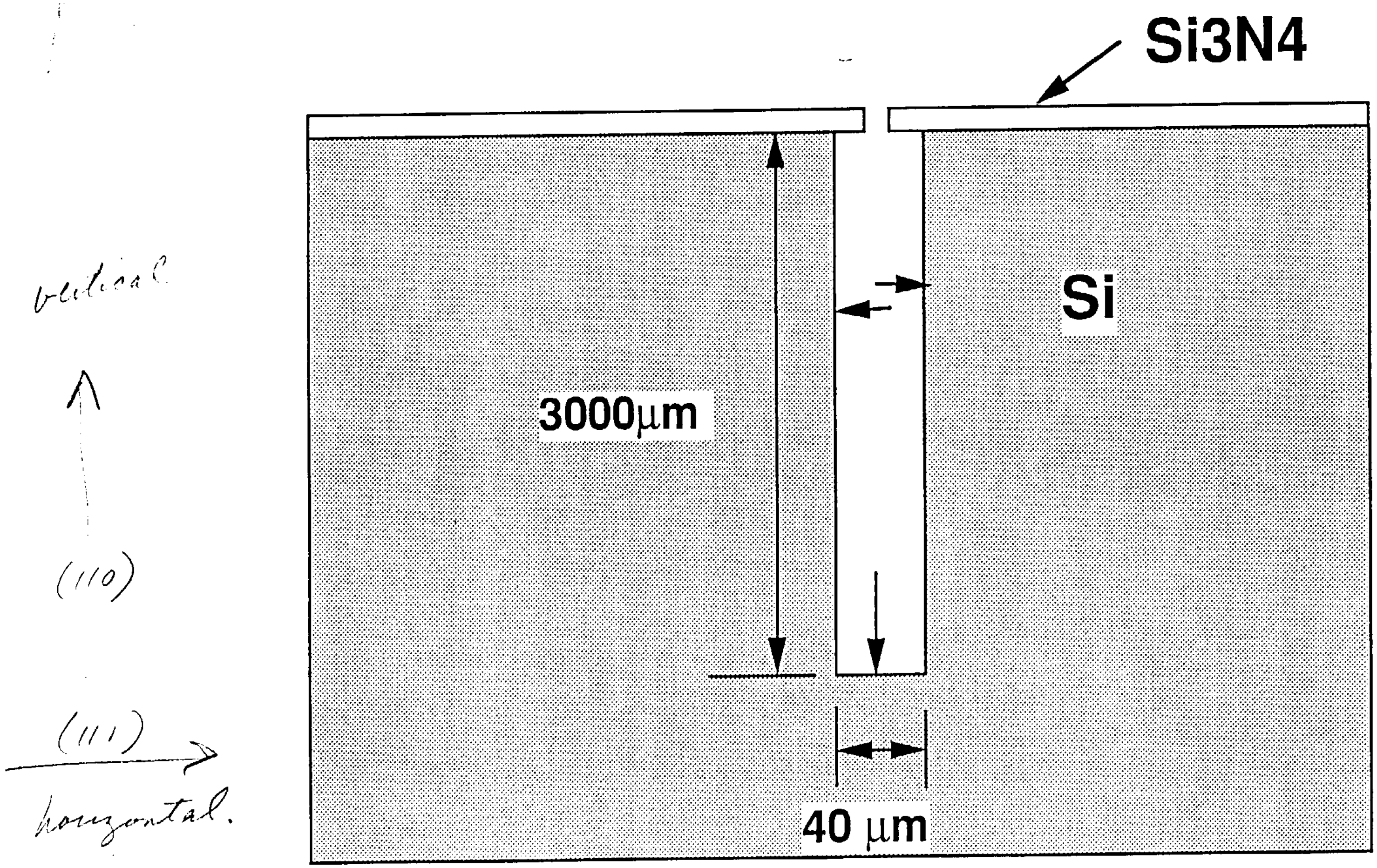


figure 1

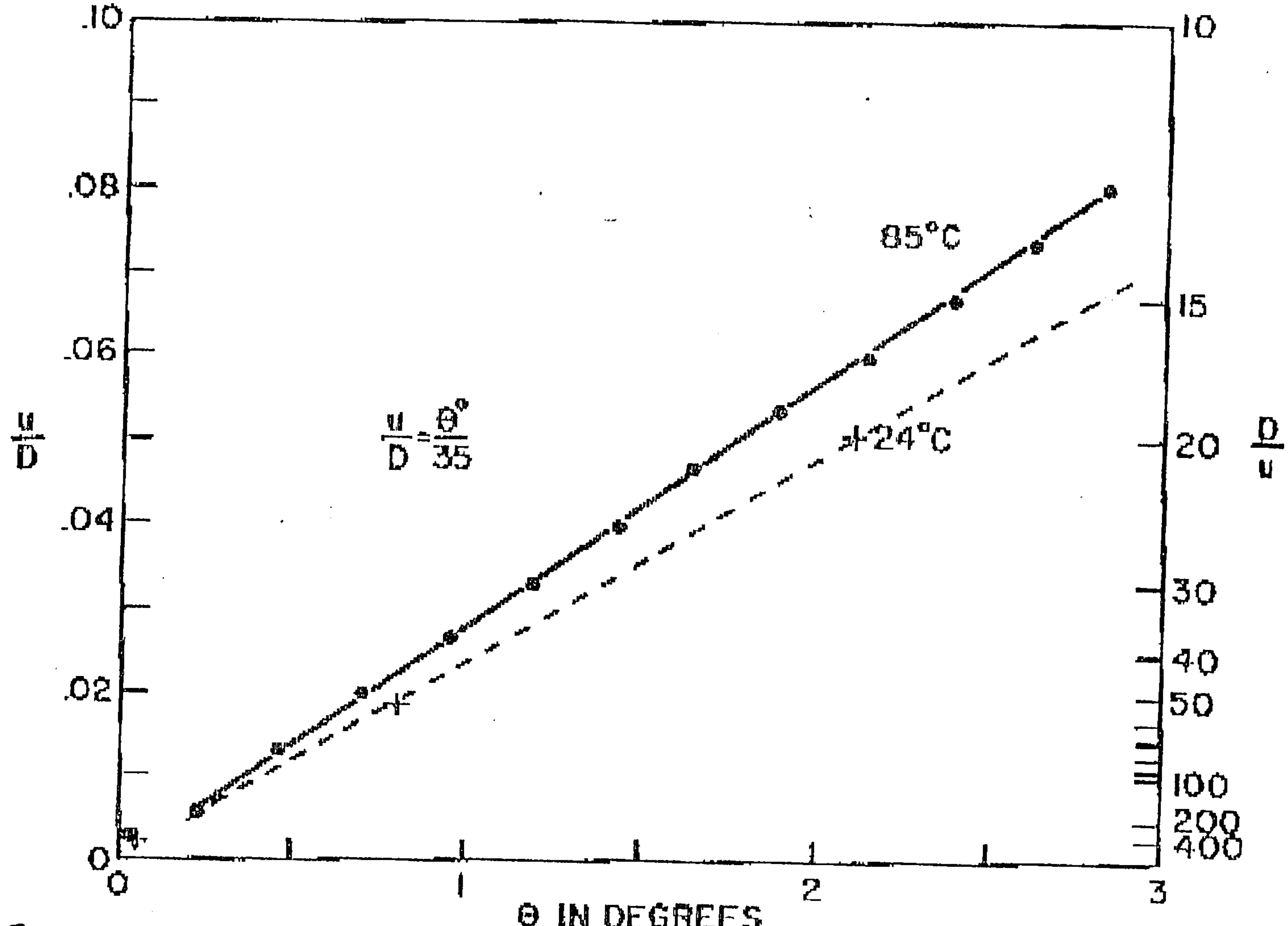


Figure 2 Lateral etching (undercutting) to depth ratio in narrow grooves at various mask misorientations in 44 wt% KOH-H_2O.

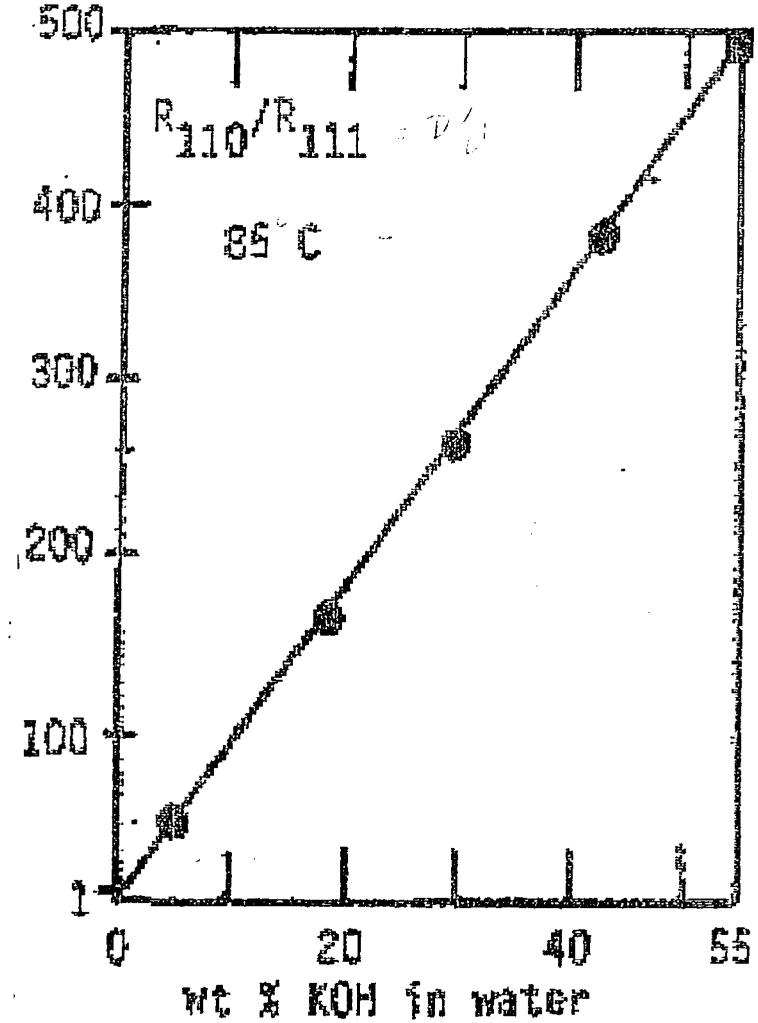
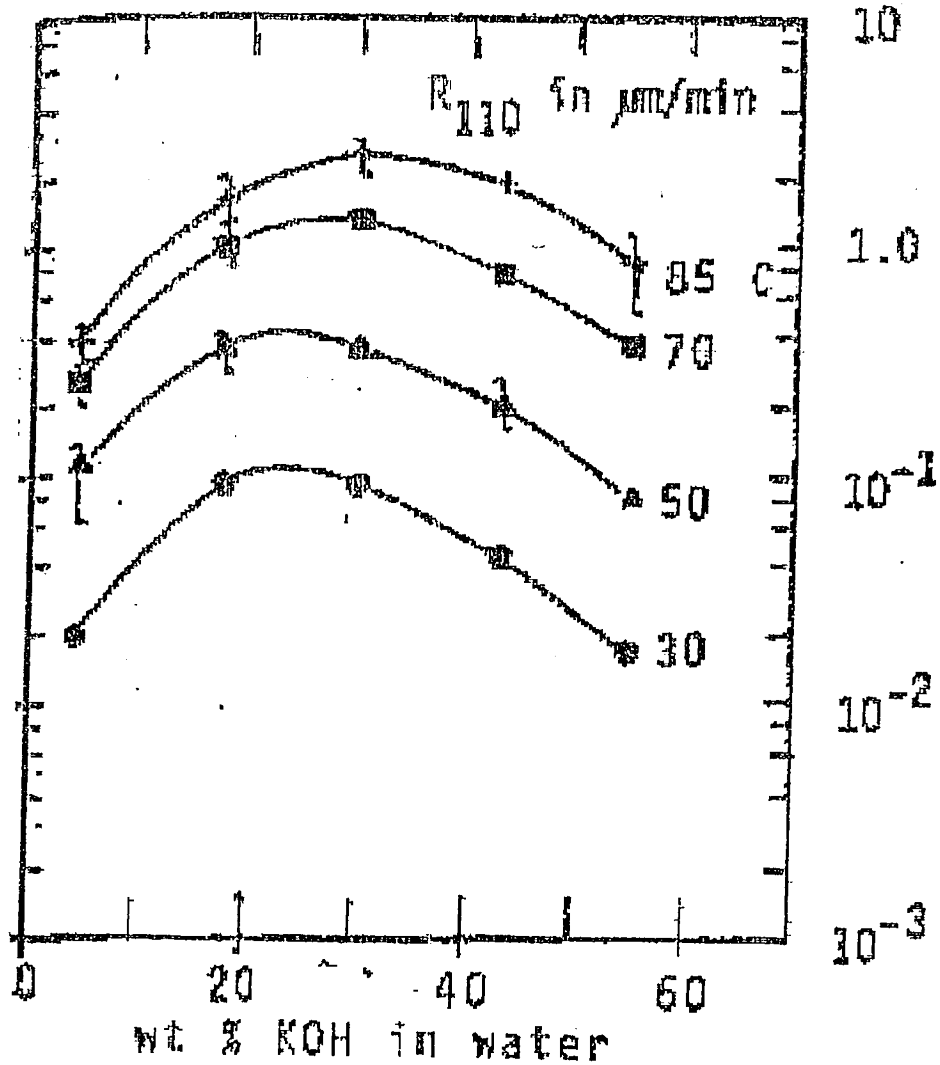
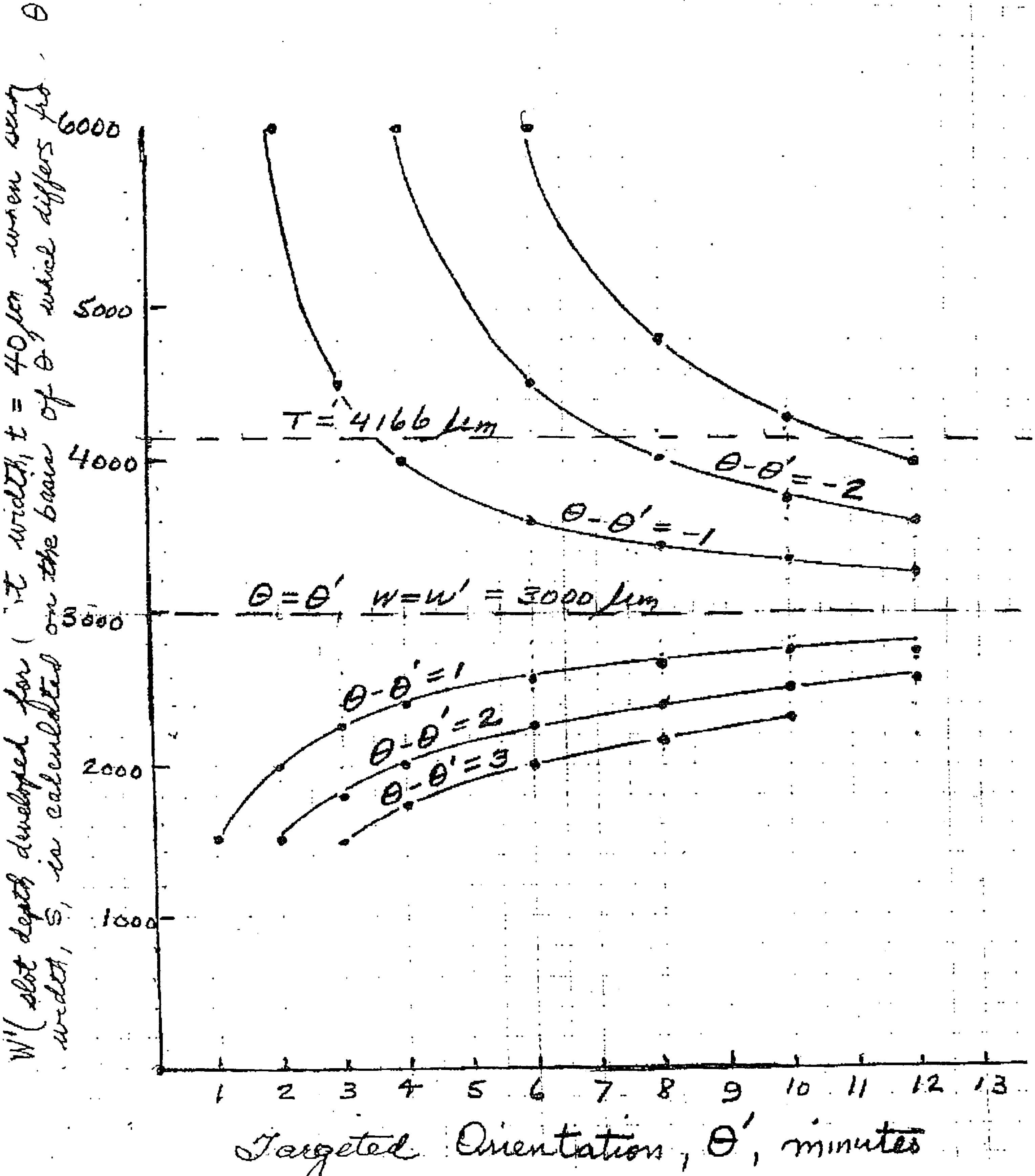


figure 3.



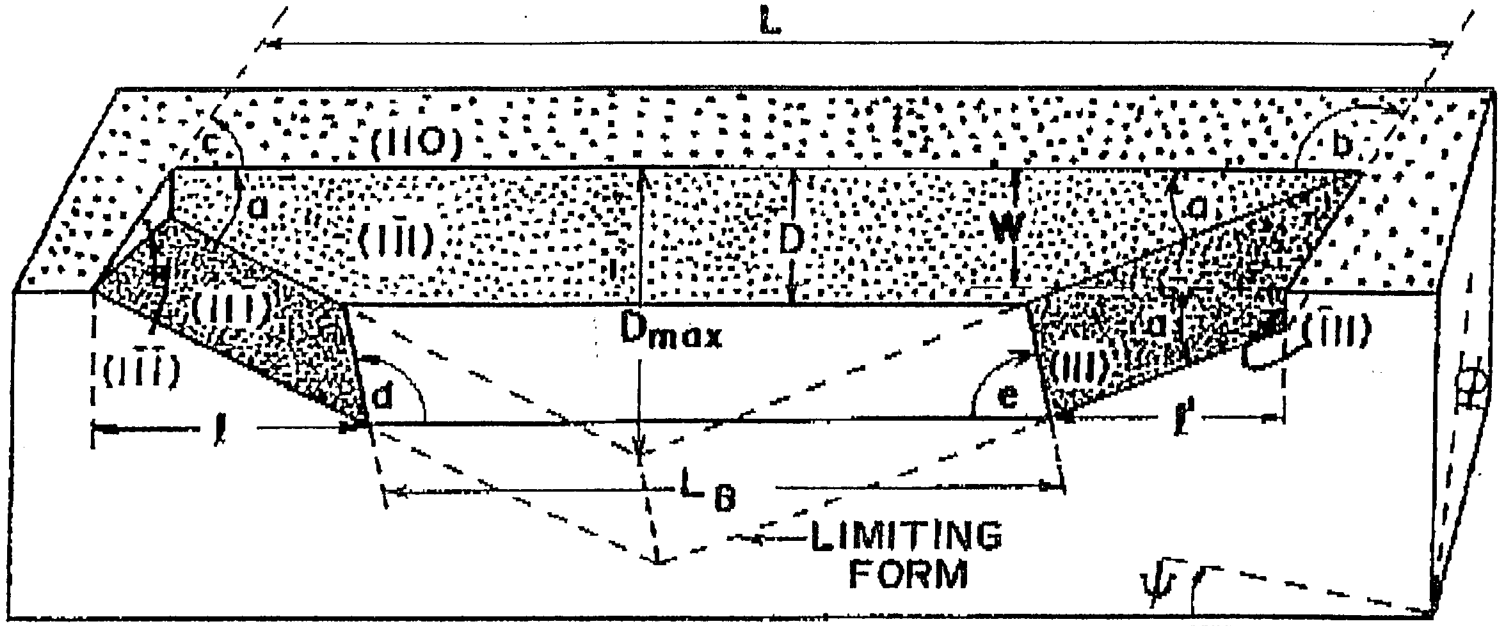
R_{110} in µm/min
85 C
70
50
30
10
1.0
10^{-1}
10^{-2}
10^{-3}
0
20
40
60
wt % KOH in water

figure 4.



W' (slot depth developed for 't width, t = 40 μm when seen
width, S, is calculated on the basis of θ' which differs fro θ
6000
5000
4000
3000
2000
1000
T = 4166 μm
θ = θ' W = W' = 3000 μm
θ-θ' = -2
θ-θ' = -1
θ-θ' = 1
θ-θ' = 2
θ-θ' = 3
1 2 3 4 5 6 7 8 9 10 11 12 13
Targeted Orientation, θ', minutes

figure 5,



$a = 30°$ $\quad$ $d = 125.26°$ $\quad$ $D_{max} = L/2\sqrt{3}$

$b = 109.5°$ $\quad$ $e = 54.74°$ $\quad$ $l = \sqrt{3}\,D$

$c = 70.5°$ $\quad$ $L_B = L - 2\sqrt{3}\,D + \frac{3\sqrt{2}\,W}{4}$ $\quad$ $l' = \sqrt{3}\,D - \frac{3\sqrt{2}\,W}{4}$

Figure 6 The geometry of the slow-etching {111} planes that develop in a long narrow groove etched into the (110) surface of Si (see text).

Incomplete grooves
A

Undercut openings
(increased width)
B

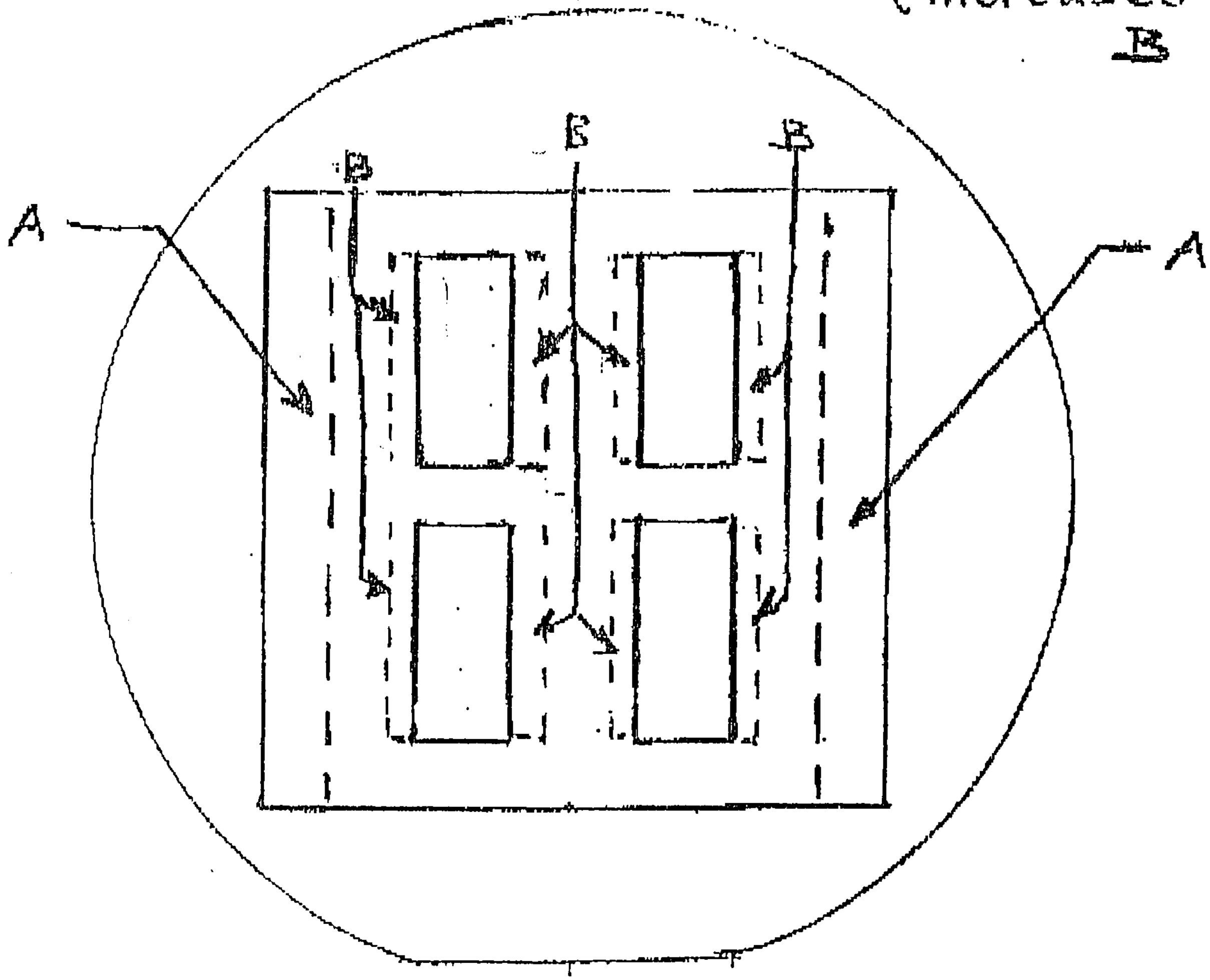


figure 7,

Interoffice Correspondence

TRW Space & Technology Group

TRW

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7/29/93

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E. N. Frazier

To:
file

cc:
GSFC

Location/Phone:
R9/1082
x20282

This memo lays out the general approach to etching the slots and open areas in the silicon wafers, taking into account all of the problems associated with undercutting as described in the memo dated 7/15/93.

<u>INTRODUCTION</u>

We start from the point where we have the 4 inch wafers fully coated with silicon nitride. In general, we will need three different masks, as shown in figure 1. The first mask is opened up in the nitride and etched while the rest of the wafer is still protected by the nitride. This first mask is the fan pattern which is used to determine the correct orientation of mask #2 with respect to the intersection of the [111] plane with the wafer surface. The diode laser group uses slots arranged at 1 arc minute intervals and can distinguish the single slot most closely oriented to the [111] plane by virtue of the minimum undercutting. We may want to design a mask with finer angular resolution (by say a factor of 2 or 4). This fan pattern mask is etched until there is measurable undercutting for all the slots. The best slot is identified and used to align mask #2.

Mask #2 defines the slots that will hold the tungsten blades. It also contains large open areas in order to leave a minimum of silicon directly over the aperture. The slots in this mask are <u>narrower</u> than the width of the desired slots in the silicon wafer to account for the undercutting that will happen. The slots are etched down the a depth which will just contain the tungsten blades, although the precise value of this depth is not critical. The open areas on the front will be etched at the same time as the slots. It is important to note at this point that we have learned from Kendall that open areas etch faster than deep slots, by a factor of 1/.7. Even with this faster etching, the open area will not be etched all the way through the wafer by the time the etching in the slots must be stopped. Therefore, we must etch the open areas from the back side as well. We will start with only mask #2, and will etch down to the point such that, when we open up the open area of mask #3 on the back side, then the open areas will etch from both sides and will meet each other when the slots have been etched down to the desired depth.

A four step process has been devised which accomplishes the slot etching and the open area etching, and accounts for the undercutting in a way which is controllable and convergent. This is sketched out in figure 2. The figure is shown to scale, except that the widths of the slots have been enlarged by 10X for clarity. The etching of the open areas is indicated only schematically (although the depths of the open areas at the different steps are shown to scale). To start with, the slots in the mask are defined to be narrower than the desired slot, and the width of this mask slot is calculated with a deliberate overestimate of the amount of undercutting. In figure 2 for example, a 15 μm mask slot is defined in order to achieve a 25 μm slot in the silicon.

In the first step, the slots are etched down a fraction of the total depth, say 1/3rd, then the wafer is removed from the etchant and the depth and width of the slot is measured. This measurement yields the true empirical value of the amount of undercutting that we must deal with.

In the second step, we calculate the difference between our estimate of the undercutting and the true undercutting, and correct for this difference by changing the concentration of the KOH etchant. In other words, we adjust the concentration of KOH such that we achieve the desired slot width at the same time that we reach the desired depth. We then return the wafer to etching and proceed until we reach the calculated point where it is time to open up the open area of mask #3 on the back side.

In the third step, we continue etching from both sides now until the open areas meet. We have started step #3 at that point such that, when the open areas meet, the depth and width of the slots will be slightly less than desired. We then take the wafer out of the etchant, and make the final measurement of slot depth and width.

The fourth step is the final "polishing" step, and can actually be repeated more than once if necessary. The objective is to widen the slot by the final small amount needed (typically a micron or so) to achieve the precise width desired, while increasing the depth only slightly. This can be done because the depth is not nearly as critical as the width. A very low concentration of KOH etchant will be used (~5%) to make the etching process as isotropic as possible.

<u>THE SPECIFIC APPROACHES</u>

We will now describe this process again, this time in more detail, and quantitatively. We will define more intermediate steps in the process and renumber the steps to account for these. This should not be confused with the numerology of the basic four step process laid out above:

<u>Desired Geometries:</u>

We will consider 4 different desired geometries, selected to cover the range from our first effort down to the finest grids presently contemplated. The starting material is a 4 inch silicon wafer, 3 mm thick. All the geometries follow the relationship:

$$(p-t)/W = 1/50 \qquad \text{where } \begin{array}{l} p = \text{pitch} \\ t = \text{slot width} \\ W = \text{slot depth} \end{array}$$

i.e. the proportions of the grids remain constant.

The four geometries are:

geometry	p,µm	t,µm	W,µm
A	60	25	1750
B	40	15	1250
C	30	12.5	875
D	25	10	750

Given these dimensions, we find that for the narrower slots, the slot depth is so small compared to the 3 mm thickness of the wafer, that we will never be able to etch the open areas, even etching from both sides, before we have to stop etching. Therefore, for these narrow slots, we have to modify the steps slightly by adding a step at the very start wherein we etch the open areas from the back side to the required depth <u>before</u> we do anything else.

Approach I. For the wider slots, the full set of steps are as follows:

1. Open mask #2 on the top surface
2. Etch a partial slot depth for evaluation of D/u
3. Calculate W', the target slot depth for front and back etches.
4. Calculate U, the target open area depth for the front etch.
5. Continue the front etch until open area depth U is attained
6. Open pattern on back surface.
7. Etch top and bottom until target slot depth W' is attained
8. Return wafer to 5% KOH solution to adjust slot width to desired value, t. Slot depth will be increased from W' to W.

Approach II. For the narrow slots:

1. Open mask #3 on the back surface
2. Etch a predetermined depth, V
3. Open mask #2 on the front surface
4. Etch a partial slot depth for evaluation of D/u
5. Calculate W', the target slot depth for the continued front and back etches.
6. Etch front and back until W' is attained
7. Return wafer to 5% KOH solution to adjust slot width to desired value t. Slot depth will be increased from W' to W. Note: For this approach, we have to choose V large enough to ensure that openings develop in the open areas of the wafer.

The final step in both approaches is for slot width adjustment. For this step, we plan to use a 5% KOH solution. This has an etching rate ratio, D/u of 40. This means that we will gain 20 μm in depth for each μm we expand the slot width. We will take this final step into consideration in choosing our target slot depth W'.

Approach I

The following is a sample calculation for geometry A; t = 25μm, W = 1750 μm. The relationship between slot width, t, slot depth W, mask slot opening, S, and misorientation, θ, is:

$$S = t - \frac{2W}{D/\mu} = 25 - \frac{3500}{2100}\,\theta \quad \text{(Kendall)}$$

Table 1 lists the mask slot widths required, S, and etching rate ratios, D/u for various misorientation angles.

θ, min.	D/u	S
1	2100	23.33
2	1050	21.67
3	700	20.0
4	525	18.33
5	420	16.67
6	350	15.0
7	300	13.33
8	262.5	11.67
9	233	10.0
10	210	8.33
11	191	6.67
12	175	5.0
13	161.5	3.33
14	150	1.67

We begin by deciding on the basis of our orientation measurements what the maximum misorientation is, and then choosing a mask slot width corresponding to a greater misorientation, i.e. a mask slot width that is narrower than needed. This is to assure that the slot width will be less that 25 µm when we attain our target slot depth, W'.

For steps 2, 5 and 7 of Approach I above, we will use identical etching conditions, for example 44% KOH at 70°C. For step 2, we will etch to an approximate depth of W/3 or about 580 µm. From the known value of S, and measured values of t_i and w_i we can calculate D/u, the etch rate ratio:

$$D/u = \frac{2W_i}{t_i - S}$$

Knowing the actual etch rate ratio, D/u, we can calculate the slot width, t, when we etch down to our target slot depth W':

$$t = \frac{2W'}{D/u} + S$$

Our target slot depth, W', is less than our final depth, W, of 1750 μm by the depth which we will gain in the final slot widening step. The target depth, W', taking this into consideration is

$$W' = \frac{W - 20(t' - S)}{1 - 40/D/u} \qquad \text{(appendix I)}$$

Example:

We chose a mask slot width, S = 15 μm, corresponding to a misorientation of 6 arc min. Actual misorientation is 3 arc min., corresponding to a D/u of 700. The required slot depth is

$$W' = \frac{1750 - 20(25-15)}{1 - 40/700} = 1643\ \mu m$$

Thus our final step will deepen our slot depth by 107 μm and widen our slot width by 107/20 or 5.35 μm.

The value W' is the desired slot depth at the end of step 7. We can use this to calculate the desired depth at the end of step 5:

U = depth of etching of the open area from the front

$$U = \left(\frac{2W'}{.7} - 3000\right) = \frac{2(1643)}{.7} - 3000 = 1694$$

After this, the back side is opened. Now etching occurs simultaneously from front and back. The total depth etched from the front = 1694 + (3000-1694)/2 = 2347 μm. On the basis of a .7 etching rate in the narrow slot, the slot depth, W' will be ; .7(2347) = 1643 μm. Thus we will attain the desired slot depth when the open areas are etched through. If the slot is not deep enough, we can return to the etching solution.

From the relationship, U = (2W'/.7 - 3000), we note that we are limited to W' > 1050μm. Thus we can use geometry B with approach I, but not geometries C and D. These with slot depths of 875 and 750 μm require Approach II.

Geometry B (Approach I):

Applying the relationship for geometry B;

$$S = t - \frac{2W}{D/u} = 15 - \frac{2500}{2100}\theta$$

Table II lists the mask slot width, S, and the etching rate ratio, D/u, for various arc minutes of misorientation.

θ, min.	D/u	S
1	2100	13.81
2	1050	12.62
3	700	11.43
4	525	10.24
5	420	9.05
6	350	7.86
7	300	6.67
8	262.5	5.48
9	233	4.29
10	210	3.10
11	191	1.91
12	175	.72

The target slot depth, W', is given by

$$W' = W - 20\left(t - \frac{2W}{D/u} - S\right) \qquad \text{(appendix I)}$$

for geometry B, t = 15 μm, W = 1250 μm

Example:

We choose a mask slot, S = 7.86 μm, corresponding to a misorientation of 6 min. Actual misorientation is 3 min., corresponding to a D/u of 700. The required slot depth, W' is

$$W' = \frac{950 + 20(7.86)}{1 - 40/700} = 1174\,\mu m$$

Thus our final step will deepen our slot depth by 76 μm and widen our slot width by 76/20 or 3.8 μm.

The value, W', the desired slot depth at the end of step 7, permits us to calculate the desired depth at the end of step 5:

$$U = \left(\frac{2W'}{.7} - 3000\right) = 354\,\mu m$$

Approach II (geometry C):

Table III lists the mask slot width required, S, and etching rate ratios for various minutes of misorientation:

θ, min.	D/u	S
1	2100	11.67
2	1050	10.83
3	700	10.83
4	525	9.17
5	420	8.33
6	350	7.50
7	300	6.67
8	262.5	5.83
9	233	5.0
10	210	4.17
11	191	3.33
12	175	2.50
13	161.5	1.67
14	150	.83

On the basis of our orientation measurements and our experience in preparing wafers with geometries A and B, we can choose S and target slot depth, W', which will require only a small amount of adjustment for slot widening in the final step.

For geometry C, W = 850 μm. Let us assume that on the basis of our measurements and experiences we choose W' = 800 μm, i.e. we expect our slot to be too narrow by 2.5 μm, requiring a final adjustment etch which will deepen the slot by 50 μm. Let V = the depth of the initial back side etch (step 2 in approach II). We can write:

$$V = T - 2W'/.7$$

$$V = 3000 - 2(800)/.7$$

$$= 714\,\mu m$$

When the front pattern is developed, the wafer is etched from the front and the back. The open area on the front will be etched (3000-714)/2 = 1143 µm. The back will also be etched an additional 1143 µm.

Total etch (open area) from the front = 714 + 1143 = 1857 µm

Total etch (open area) from the back = 1143 µm

Total etch, front and back = 1857 + 1143 = 3000µm

The total etch of the slots will be; .7(1143) = 800µm. The slots will be widened with continued etching in 4% KOH and the slots will be deepened by 50 µm.

Approach II (geometry D):

Table IV lists the slit widths, S, and etching rate ratios, D/u for various misorientations, q:

θ, min.	D/u	S
1	2100	9.29
2	1050	8.57
3	700	7.86
4	525	7.14
5	420	6.43
6	350	5.71
7	300	5.00
8	262.5	4.29
9	233	3.57
10	210	2.86
11	191	2.14
12	175	1.43
13	161.5	0.71
14	150	-0-

For geometry D, W = 750 µm. Assume W' = 700 µm. Then;

$$V = T - 2W'/.7 = 3000 - \frac{1400}{.7} = 1000\ \mu m$$

Having etched 1000 µm from the back on the initial etch, we will etch an additional 1000 µm from both front and back on the second etch. Thus a total of 3000µm will be etched in the open area.

Total etch (open area) from the front = 1000 µm

Total etch of the slots = .7(1000) = 700µm

Appendix I

$$D/u = \frac{r_{110}}{r_{111}} = \frac{2W'}{t'-S}$$

where t' = actual slot width corresponding to actual slot depth, W'

S = mask slot width

t = target slot width

During the final etching adjustment, the slot width is increased from t' to t, and the slot depth is increased from W' to W. For 5% KOH used the final etching;

$$\frac{W-W'}{t-t'} = 20; \qquad \frac{D}{u} = \frac{2W'}{t'-S}$$

$$2(t-t') = 20\left(t - \frac{2W'}{D/u} - S\right) = W - W'$$

$$W' = W - 20(t-S) + \frac{40\,W'}{D/u}$$

$$W'\left(1 - \frac{40}{D/u}\right) = W - 20(t-S)$$

$$W' = \frac{W - 20(t-S)}{1 - 40/D/u}$$

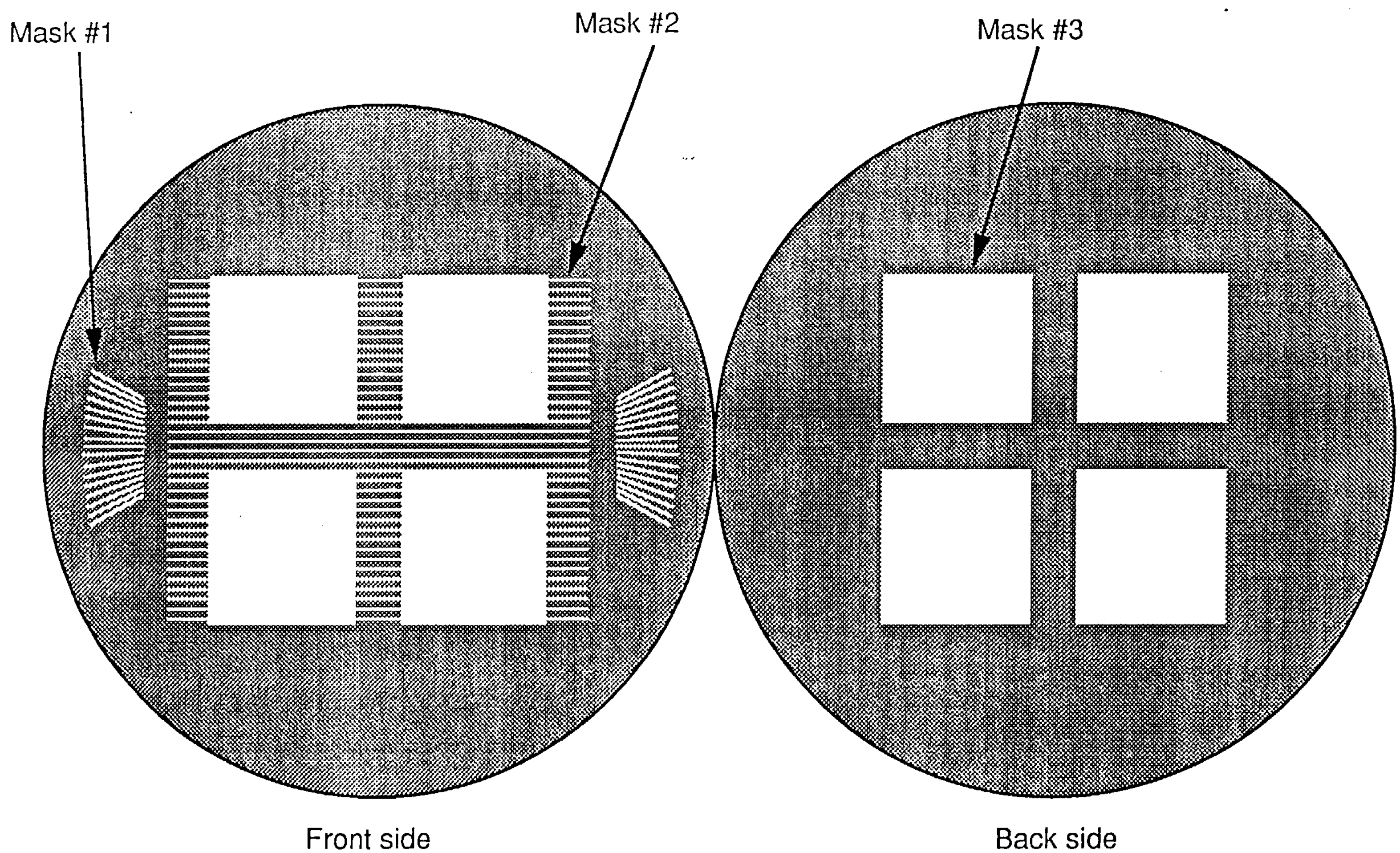

FIGURE 1. THE THREE MASKS REQUIRED

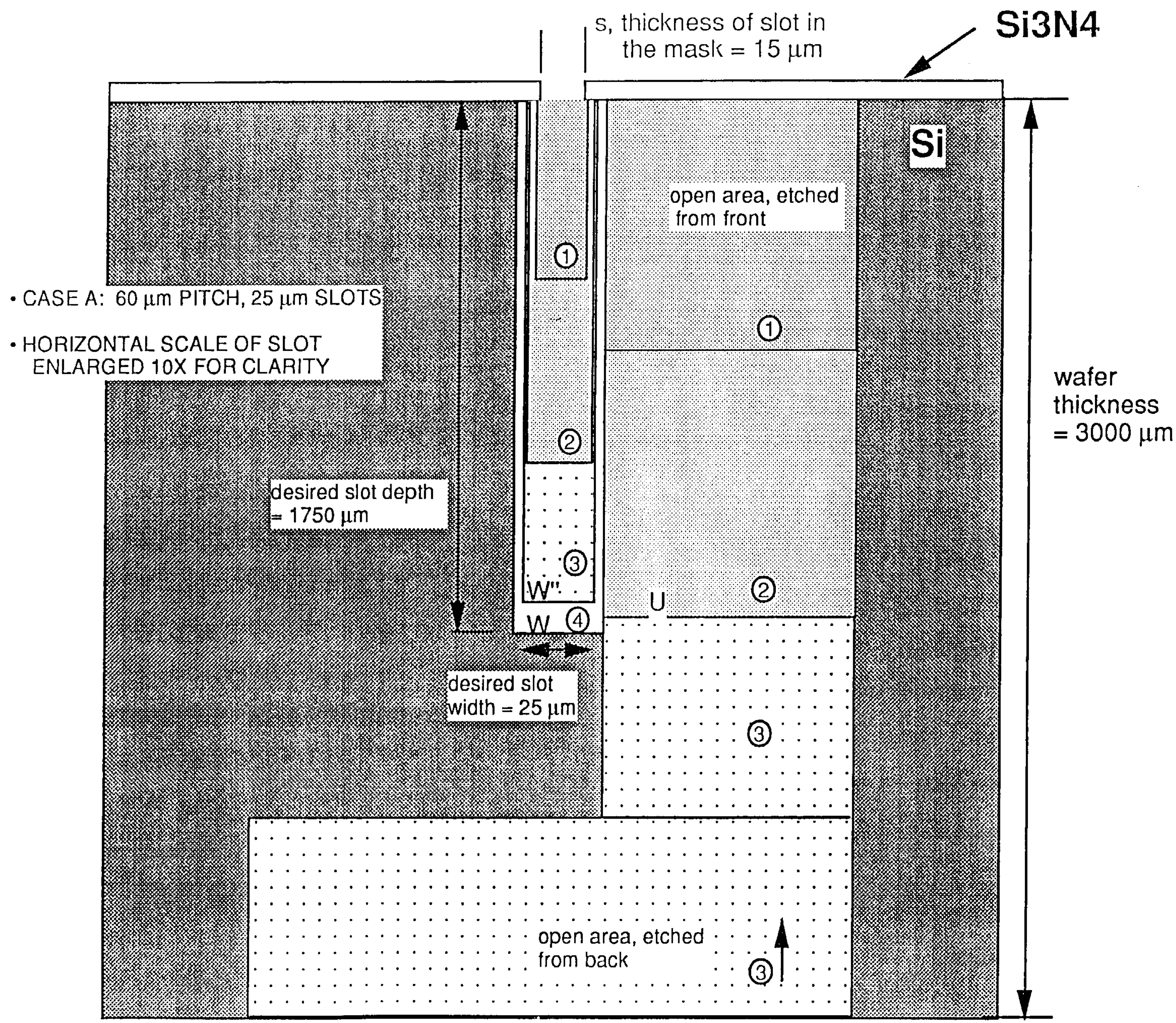

FIGURE 2. THE FOUR ETCHING STEPS

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There is a need to develop fine pitch x-ray collimator grids as an enabling technology for planned future missions. The grids consist of an array of thin parallel strips of x-ray absorbing material, such as tungsten, with pitches ranging from 34 microns to 2.036 millimeters. The grids are the key components of a new class of spaceborne instruments known as "x-ray modulation collimators." These instruments are the first to produce images of celestial sources in the hard x-ray and gamma-ray spectral regions.

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